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The Microstructure of Zinc Coatings



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The Microstructure of Zinc Coatings

By
W. H. Finkeldey

Research Division
The New Jersey Zinc Company

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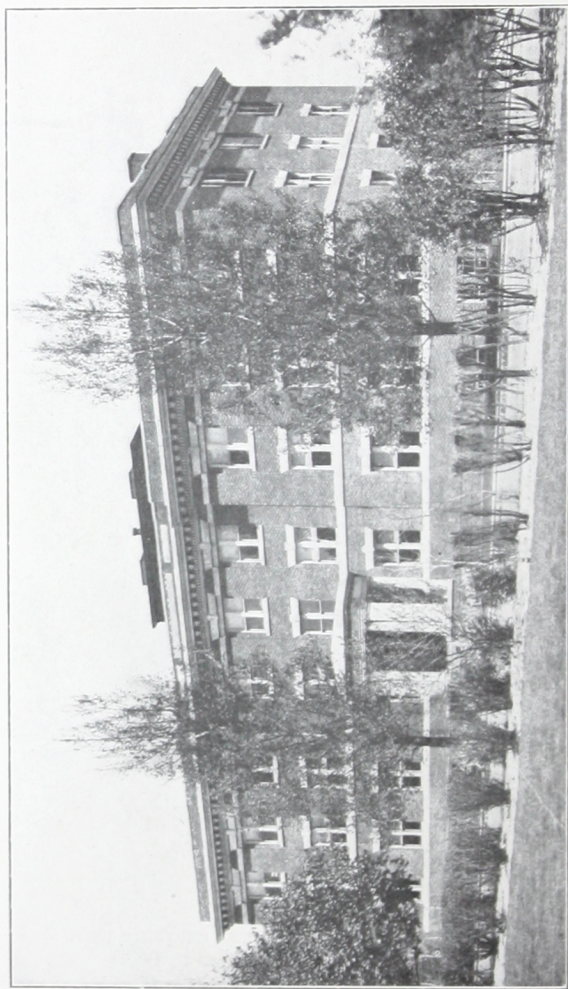
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THE MICROSTRUCTURE OF ZINC COATINGS¹

BY WILLIAM H. FINKELDEY²

SYNOPSIS

This paper describes the preparation of specimens of galvanized materials for microscopical examination. Under this head is included the various methods of mounting specimens so they may be properly sectioned and polished, also the technique of etching which has been found to give the best results.

There is some discussion of the structural constituents which make up the coating, both from the standpoint of what one might expect from the iron-zinc diagram and also on the basis of what can be actually seen in the average microsection. The reader is cautioned against the too literal use of the terms FeZn_3 and FeZn , until more is known about the exact composition of the various iron-zinc constituents and a method developed which will permit their accurate identification under the microscope.

A number of photomicrographs are given illustrating the types of zinc coatings produced by the various hot galvanizing processes now in use in coating wire and sheet. The distortion, cracking and flaking, which such zinc coatings undergo when they are severely deformed, is also illustrated by photomicrographs. The various factors in the galvanizing process, which have an effect on the type of zinc coating produced, are enumerated. A few general observations on the relation of microstructure to the bending properties of zinc coatings are also given.

Photomicrographs are included to show the types of zinc coatings produced by the electrogalvanizing, sherardizing or sprayed zinc process.

The application of protective metallic zinc coatings to iron and steel has been practiced commercially for almost a century and the microstructure of metals has been studied for the past fifty years, but it is only within comparatively recent times that the microstructure of galvanized coatings has received much attention. Although Rawdon,³ Raydt and Tammann,⁴ Arthur and Walker,⁵ Guertler⁶ and

¹ A contribution from the Research Laboratories of the New Jersey Zinc Co.

² Assistant to Chief of Research, Research Division, New Jersey Zinc Co., Palmerton, Pa.

³ H. S. Rawdon, "Structure of Commercial Zinc Coatings," *Proceedings, Am. Soc. Testing Mats.*, Vol. XVIII, Part I, p. 216 (1918).

⁴ V. Raydt and G. Tammann, "Structure and Properties of Zinc-Iron Alloys Melted Under Pressure," *Zeitschrift für Anorganische Chemie*, Vol. 83, p. 257 (1913).

⁵ W. Arthur and W. H. Walker, "Structure of Galvanized Iron," *Am. Inst. of Metals*, Vol. 6, p. 82 (1912).

⁶ W. Guertler, "Structure of Galvanized Iron," *Zeitschrift für Metallographie*, Vol. 1, p. 353.

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others have all made valuable contributions to our fund of information on this subject, there still remains a large field to be explored.

It is with the hope of making further additions to this general store of knowledge that the present paper has been written. However,

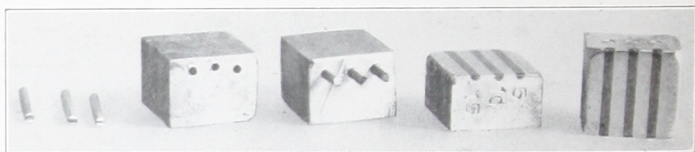


FIG. 1.—Soft Zinc Mount for Galvanized Wires.

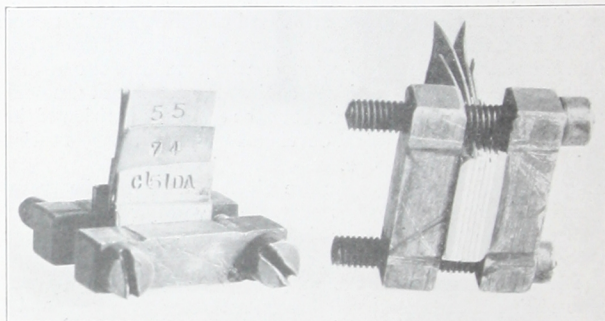


FIG. 2.—Galvanized Sheets Between Sheets of Soft Zinc Mounted in Clamp.



FIG. 3.—Mold Used in Casting Mounts With Molten Zinc. Mount Before and After Sectioning and Polishing.

in its preparation a number of points have arisen which cannot be satisfactorily explained. The far-reaching importance of these unanswered questions suggests that in this particular field of metallography our greatest advances are still to be made.

PREPARATION OF MICROSECTIONS

In preparing samples of zinc-coated materials for examination under the microscope, it is absolutely essential that some form of mounting be used which will reinforce and protect the outside edge of the coating during the polishing operation. This can be accomplished in a number of ways; two of the best are illustrated in Figs. 1, 2 and 3. In one case, mounts of pure soft zinc in the form of rolled bars or thin sheets are slowly flowed into intimate contact with the surface of the zinc coating by pressure applied by a vise and clamps. In the other, the specimens are placed in a small iron mold secured in the proper position, and molten zinc, at a temperature just above the melting point, is poured around them.¹ The first method is not well adapted for the mounting of bent galvanized wires or sheets or objects of irregular shape; the second method has the disadvantage of heating the zinc coating, which, however, in most cases does not prevent the use of the method.

After mounting, the specimens are sectioned by sawing and leveling off with a file. They are then rubbed down first on No. 2 carborundum cloth followed by No. 0, No. 00, and No. 000 Huberts' French emery papers. The wet polishing is done on the usual polishing wheels in three steps: (1) SFX emery powder on a good grade of white duck canvas; (2) 6X emery powder on broadcloth; (3) 10X emery on broadcloth. The emery powders are first shaken up in a bottle with water and applied to the wheels in the form of a thin mud. A moderate wheel speed and plenty of water should be used. It is not advisable to attempt to polish out all the scratches in the steel base, since the shortest possible polishing time is desired in order to reduce the inevitable difference in level between the soft zinc coating and the hard steel base. For the same reason, if it is possible, it is well to avoid having the scratches parallel the line of contact between the steel base and the coating at any time during the polishing operations.

Before etching, the specimens should be thoroughly washed first in ether, then in alcohol, and finally, in water. After shaking or blowing off the excess water from the surface of the specimen, the specimen should be immersed and agitated for about eight seconds in the following etching solution: 100 cc. water, 20 g. c. p. chromic acid (free from sulfates), 1.5 g. sodium sulfate (anhydrous). When the specimen is removed from the etching solution it should be *immediately* washed in a stream of running water, care being taken

¹ This method of mounting was first demonstrated to the author by Mr. F. M. Crapo, Chief Engineer of the Indiana Steel and Wire Co.

that the stream impinges first on the polished face of the mount. Enough etching solution should be used in the case of large mounts and specimens mounted in clamps so that the polished face is totally immersed at all times; otherwise disturbing tarnish films may be developed over the surface of the specimens.

STRUCTURAL CONSIDERATIONS (HOT-DIPPED GALVANIZED COATINGS)

The constitutional diagram of zinc-iron alloys, as given by Raydt and Tammann,¹ and further amplified by Peirce,² can be made to account for the existence of a number of structural fields or constituents which might be considered to be present in a galvanized coating produced by the hot-dip process. First, there is iron containing zinc in solid solution, second, an iron-zinc constituent having a composition corresponding to FeZn_3 , third, a phase whose composition varies from 10.9 per cent of iron (FeZn_7) to 7.5 per cent of iron and finally there is zinc with not more than 0.01 per cent of iron in solid solution.

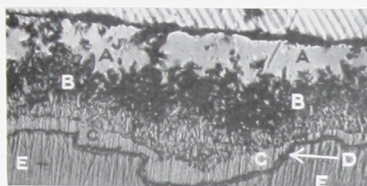
It is questionable just how much we are justified in interpreting what the microstructure reveals in terms of the constitutional diagram. In the determination of such diagrams careful consideration must be given to temperature control and the allowance of sufficient time for equilibrium to be reached at every given set of conditions. This practice can hardly be said to be duplicated in the ordinary commercial "hot-dip" galvanizing process. Furthermore, we do not have as yet means of definitely identifying either of the constituents FeZn_3 or FeZn_7 .

The author prefers to consider the various structural features on the basis of only what can be seen when a micro-examination is made. Referring to Fig. 4 it is possible to distinguish the following components of the coating:

1. A layer *A* of relatively pure zinc.
2. A fairly wide band *B* of duplex structure consisting of a hard, zinc rich, iron-zinc constituent more or less dispersed in a matrix of *A*.
3. A narrower layer *C* consisting entirely of a zinc rich iron-zinc constituent, hard, crystalline and light colored.
4. A very thin layer *D* which is also a hard, crystalline, iron-zinc constituent, darker colored and less rich in zinc than *C*.
5. Finally *E* the steel base.

¹ *Loc. cit.*

² W. M. Peirce, "Studies on the Constitution of Binary Zinc Base Alloys," *Transactions, Am. Inst. Mining and Metallurgical Engrs.*, Vol. 68, p. 772.



- A.—Relatively pure zinc.
 B.—Duplex structure, hard iron zinc constituent dispersed in matrix of A.
 C.—Iron-zinc constituent, hard crystalline, light colored.
 D.—Iron-zinc constituent, also hard and crystalline. Less rich in zinc and darker colored than C.
 E.—Steel base.

FIG. 4.—Heavy Zinc Coating on Steel Sheets, Produced by "Hot-Dip" Process, Showing Four Constituents.



FIG. 5.—Typical of heavy coatings (2-oz. per sq. ft. of sheet) on pure iron and low carbon steel.



FIG. 6.—Coating on medium carbon steel.

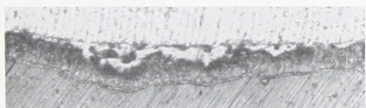


FIG. 7.—Commercial (1.25 oz.) coating on pure iron sheet.

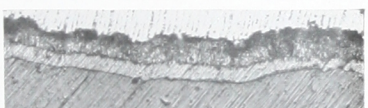


FIG. 8.—Same sheet shown in Fig. 7 after heating for 2 hours at 350° C.

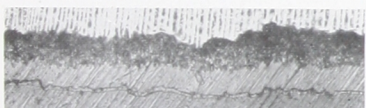


FIG. 9.—Same as Fig. 7 after heating for 4 hours at 350° C.

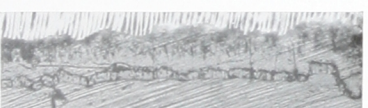


FIG. 10.—Same as Fig. 7 after heating for 5 hours at 350° C.



FIG. 11.—Light weight coating (1.2 oz.) on mild steel base, suitable for forming operations.

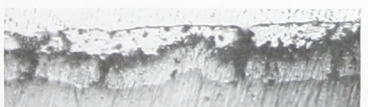


FIG. 12.—Same sheet as Fig. 11 after right angle bend with $\frac{1}{4}$ in. radius fillet. Cracking, but no flaking.

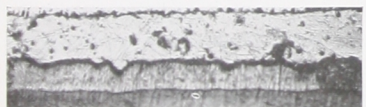


FIG. 13.—Medium heavy coating (1.5 oz.) on mild steel base.

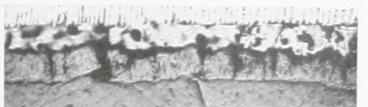


FIG. 14.—Same sheet as Fig. 13, after right angle bend with $\frac{1}{4}$ in. radius fillet. Cracking with peeling of coating from base.

FIGS. 4-14.—Photomicrographs of Hot-Dip Galvanized Coatings. ($\times 350$, Reduced One-third in Reproduction.)

It is seldom that all of the foregoing structural features are easily distinguishable in one section as shown in Fig. 4. Microstructures similar to those shown in Figs. 5, 6 and 7 are more often encountered in the examination of ordinary commercial hot galvanized coatings on iron or steel sheets.

The free use of the terms FeZn_3 and FeZn_7 to designate the alloy constituents of a galvanized coating is open to some criticism if the presence of a definite chemical compound is implied. For example, in structures similar to that shown in Fig. 5 the wide band of alloy is frequently designated as FeZn_7 and the very narrow band next to the steel base as FeZn_3 . While admitting that this may be the correct interpretation of the structure, it is the author's belief, at least in reference to this particular section, that the narrow band is an iron zinc compound probably of the composition FeZn_7 while the darker colored wide band of alloy is the duplex structure *B* previously referred to. The columnar-like crystals of iron-zinc constituent in this layer may closely approach the composition FeZn_7 at their base and become less rich in iron as they grow out into the matrix of zinc.

The evidence supporting this belief is shown in Fig. 4 and also in the microstructures secured by heating a galvanized coating similar to that shown in Fig. 5. Samples of commercial hot-galvanized sheet were heated for various lengths of time at a temperature of 350°C . in a container filled with zinc dust to cut down surface oxidation of the coating. Under these conditions the alloy layers will grow as iron diffuses into the coatings. Fig. 7 shows the original sheet before heating and Figs. 8, 9 and 10 show the growth of the alloy constituents at various stages during the heating cycle. In Figs. 9 and 10 a narrow band of alloy next to the steel can be seen which was not discernible in the original structure before heating. This is believed to be the compound FeZn_3 . It may be present in every galvanized coating produced by the hot-dip process but not always developed to a sufficient extent to be easily recognized with our present technique of polishing and etching.

A satisfactory answer of this question probably will not be given until such a time as we have developed a metallographic technique which permits the positive identification of the compounds FeZn_3 and FeZn_7 .

On the basis of convenience there is some justification for the use of the terms FeZn_3 and FeZn_7 to indicate in general the iron-zinc ratio and it is in this sense that they are used throughout the rest of this paper.

MICROSTRUCTURE VERSUS PHYSICAL PROPERTIES OF ZINC COATING

There is every reason to believe that the physical properties of the coating, so far as its resistance to corrosion and its malleability is concerned, are related to its microstructure. Messrs. Hocker and Farnsworth¹ have already shown that the rate of corrosion of hot-dipped galvanized coatings depends to a great extent upon the presence and amount of certain iron-zinc alloys. Unfortunately, all the relations existing between the microstructure and the bending properties of the zinc coating are not easily explained and in many cases not even known. There are, however, a number of factors, whose significance we are just beginning to appreciate, which have a marked influence on both the type of microstructure produced and the bending properties of the coating.

It is becoming more and more evident that the conditions existing at the surface of the steel after the heat treating, pickling and fluxing operations preparatory to galvanizing are completed have a marked effect on the type of microstructure that results. The chemical composition of the steel is also an important factor, both in respect to its influence on the type of surface produced by pickling operations as well as the probable influence that the carbon, phosphorus, sulfur, etc., in the steel have on the growth of alloy layers. This is illustrated in part by the microstructures shown in Figs. 5 and 6 and also Figs. 19 and 29. Apart from the effects produced by the foregoing variables, the type of microstructure produced is also dependent on the temperature of the zinc bath and the length of time the article remains in it during the galvanizing operation (see Fig. 17(b)). The composition of the bath is likewise an important factor. In addition, in the case of wire, the type of structure will depend upon the treatment the wire receives after it leaves the zinc bath, that is, whether it is drawn through charcoal (so-called double galvanized or unwiped wire) or through an asbestos wipe under pressure, or unwiped and heat treated. The space limitations of this paper do not permit a more detailed discussion of all these points. However, the effects of several of the variables, cited in the foregoing paragraph, are illustrated in a number of the photomicrographs.

A few general observations on the relation of microstructure to bending properties can be made which hold true in a large majority of the cases:

1. In general the thicker the zinc coating the more liable it is to peel when subjected to severe deformation (see Figs. 13, 14, 25 and

¹ An informal paper presented at the general meeting of Committee A-5, March 18, 1926, Providence, R. I.

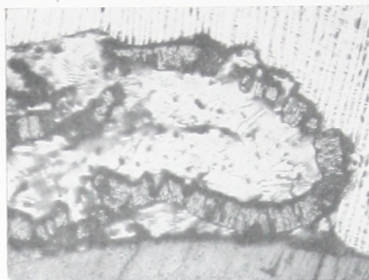


FIG. 15.—Same sheet as Fig. 13, showing bad peeling of the coating after severe forming.

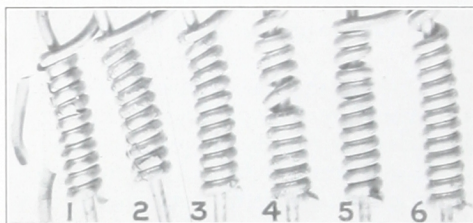


FIG. 18.—No. 12 gage wires wrapped on own diameter to test adherence and malleability of zinc coating; specimens arranged in order poorest No. 1 to best No. 6.



FIG. 19.—E.B.B. zinc-coated wire. No. 1 in Fig. 18.

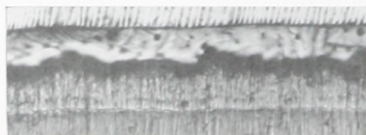


FIG. 21.—Mild steel zinc-coated wire. No. 2 in Fig. 18.

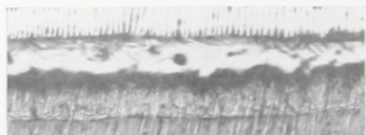


FIG. 23.—B.B. zinc-coated steel wire. No. 3 in Fig. 18.

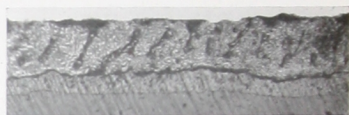


FIG. 16.—Coating high in tin, diagonal streaks in pure zinc part of coating tin-zinc eutectic.

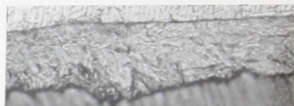


FIG. 17(a).—Coating produced with 0.2 per cent aluminum in zinc bath. No visible alloy layers.

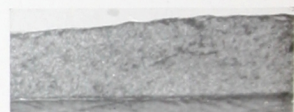


FIG. 17(b).—Wire galvanized in $\frac{1}{10}$ sec. Experiment to show time factor on growth of alloy.



FIG. 20.—Same wire as Fig. 19, wrapped on mandrel 3 times diameter of wire. Bad cracking and peeling.

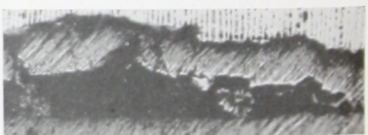


FIG. 22.—Same wire as Fig. 21, wrapped on mandrel 3 times diameter of wire. Coating flaking.

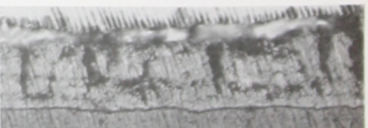


FIG. 24.—Same wire as Fig. 23, wrapped on mandrel 3 times diameter of wire. Coating cracked.

FIGS. 15-24.—Photomicrographs of Hot-Dip Galvanized Coatings. ($\times 350$, Reduced One-third in Reproduction.)

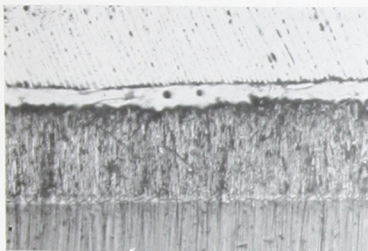


FIG. 25.—Bessemer steel wire, very heavy zinc coating 1.5 oz. per sq. ft. of surface. No. 4 in Fig. 18.

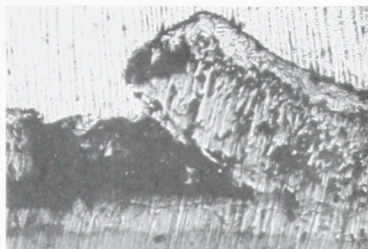


FIG. 26.—Same wire as Fig. 25, wrapped on own diameter. Note separation between alloy layers.

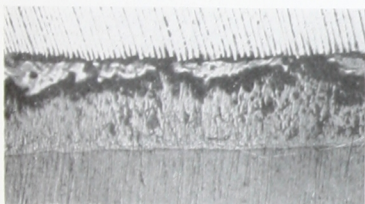


FIG. 27.—Zinc coating on 30-40 per cent carbon steel wire. No. 5 in Fig. 18.

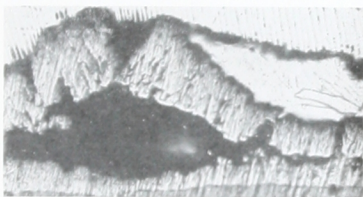


FIG. 28.—Same wire as Fig. 27, wrapped on own diameter. Coating checks, but no flaking. Note some alloy still adhering to steel base.

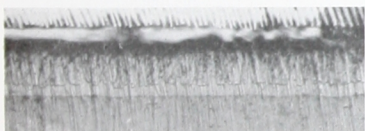


FIG. 29.—Zinc coating on 50-60 carbon steel wire. No. 6 in Fig. 18.



FIG. 30.—Same wire as Fig. 29, wrapped on own diameter. Coating shows fine hair cracks. Note cracks penetrate first alloy layer only.

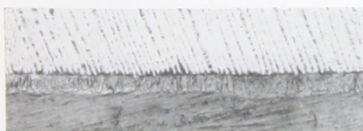


FIG. 31.—"Tight" wiped wire coating (0.26 oz.) composed entirely of zinc-iron alloy.

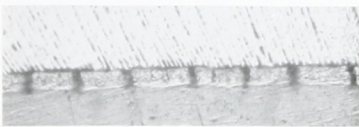


FIG. 32.—Same wire as Fig. 31, wrapped on own diameter. Cracking, but no flaking.

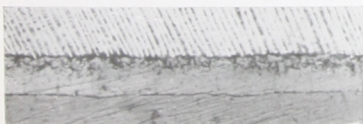


FIG. 33.—Asbestos wiped wire, coating (0.65 oz.) all zinc-iron alloy.

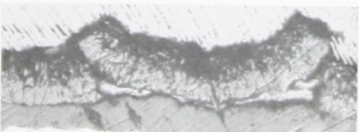


FIG. 34.—Same wire as Fig. 33, wrapped on own diameter. Cracking, some flaking.

FIGS. 25-34.—Photomicrographs of Hot-Dip Galvanized Coatings. ($\times 350$, Reduced One-third in Reproduction.)

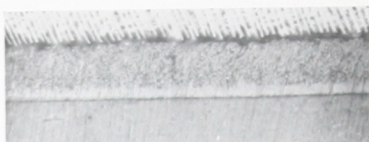


FIG. 35.—Type of coating secured with unwiped hot galvanizing followed by heat treatment. Coating (0.6 oz.) all iron-zinc alloy.

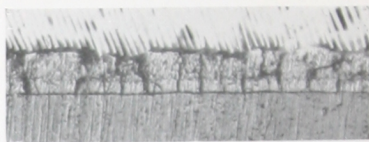


FIG. 36.—Same wire as Fig. 35, wrapped on own diameter. Many fine cracks, but no peeling or flaking.

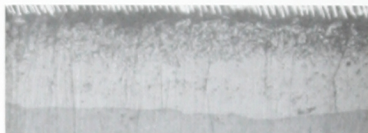


FIG. 37.—Same type wire as Fig. 35. Coating (0.7 oz.) all iron-zinc alloy.

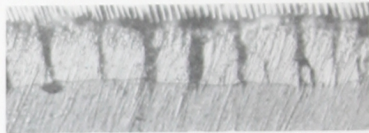


FIG. 38.—Same wire as Fig. 37, wrapped on own diameter. Many fine cracks, no peeling or flaking.

FIGS. 35-38.—Photomicrographs of Hot-Dip Galvanized Coatings. ($\times 350$, Reduced One-third in Reproduction.)

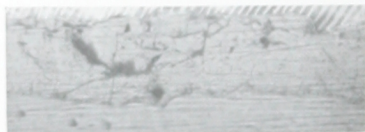


FIG. 39.—Zinc coating produced by the sherardizing process. All zinc-iron alloy 0.9 oz. per sq. ft. of surface. ($\times 350$, reduced one-third in reproduction.)



FIG. 40.—Electro-deposited coating. Zinc coating 0.86 oz. per sq. ft. of surface. No alloy. ($\times 100$, reduced one-third in reproduction.)

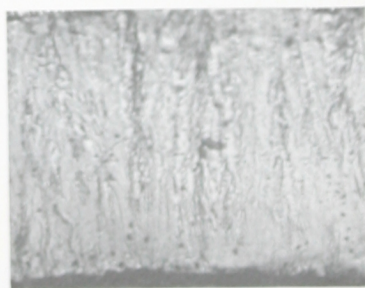


FIG. 41.—Same as Fig. 40. ($\times 500$, reduced one-third in reproduction.)

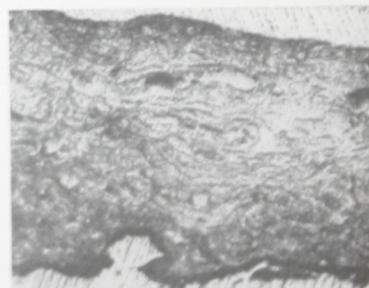


FIG. 42.—Sprayed (Schoop Process) zinc coating, very irregular, average 0.97 oz. per sq. ft. of surface. No visible alloy; note inclusions of oxide.

FIGS. 39-42.—Photomicrographs of Zinc Coatings Produced by the Sherardizing, Electro-Galvanizing and "Schoop" Spray Processes.

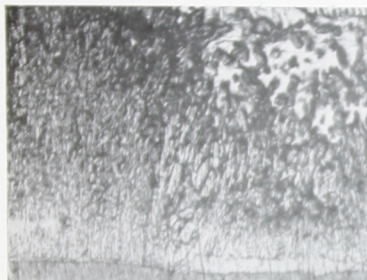


FIG. 43.—Steel, Carbon 0.11 per cent, Phosphorus 0.28 per cent, Sulfur 0.058 per cent, Silicon 0.061 per cent, Manganese 0.55 per cent, Copper 0.040 per cent.

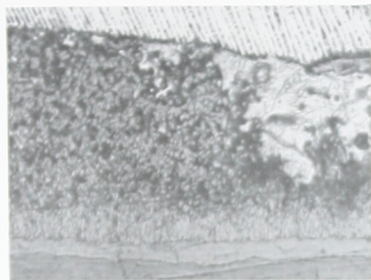


FIG. 44.—Steel, Carbon 0.11 per cent, Phosphorus 0.028 per cent, Sulfur 0.059 per cent, Silicon 0.056 per cent, Manganese 0.55 per cent, Copper 0.040 per cent.

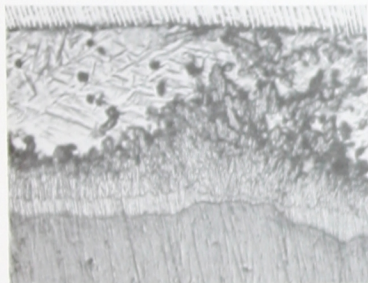


FIG. 45.—Steel, Carbon 0.20 per cent, Phosphorus .008 per cent, Sulfur 0.030 per cent, Silicon 0.019 per cent, Manganese 0.59 per cent, Copper 0.012 per cent.

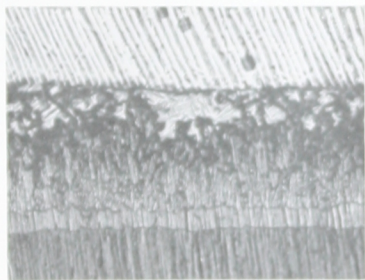


FIG. 46.—Steel, Carbon 0.21 per cent, Phosphorus 0.102 per cent, Sulfur 0.030 per cent, Silicon 0.061 per cent, Manganese 0.68 per cent, Copper 0.032 per cent.

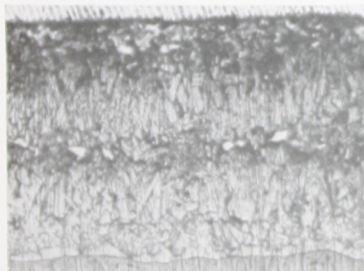


FIG. 47.—Steel, Carbon 0.36 per cent, Phosphorus 0.011 per cent, Sulfur 0.028 per cent, Silicon 0.131 per cent, Manganese 0.52 per cent, Copper 0.012 per cent.

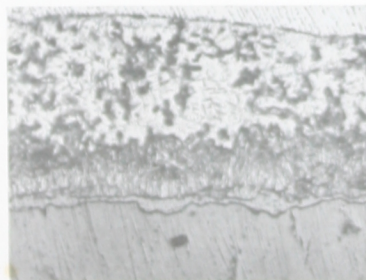


FIG. 48.—Pure iron, Carbon 0.027 per cent, Phosphorus 0.008 per cent, Sulfur 0.049 per cent, Silicon 0.01 per cent, Manganese 0.008 per cent.

FIGS. 43-48.—Photomicrographs of Zinc Coatings Produced by Hand-Dip Galvanizing with Steels of Various Compositions. ($\times 350$, Reduced One-third in Reproduction.) Bars, $\frac{1}{4}$ by 2 by 18 in.

26). This is true even with asbestos wiped wires consisting almost entirely of iron-zinc alloy (see Figs. 33 and 34). Conversely, thin coatings in general have good bending properties (see Figs. 11, 12, 31 and 32). However, by carefully controlling certain conditions of the galvanizing process, heavy coatings can be produced on wires which will withstand severe bending.

2. An irregular alloy layer, similar to that shown in Fig. 5, is usually associated with poor bending properties, and a smooth even alloy layer (see Fig. 7) with good bending properties. An irregularity in the line of contact between the steel and the alloy layers is also suspected of producing poor bending properties.

3. The presence of cadmium and lead in the zinc bath influences, to a marked extent, the ductility of the pure zinc part of the coating. This point is of great importance in securing ductile coatings on the heavily coated (one ounce per square foot of surface) wires galvanized by the "charcoal wiped" process. So far as the author knows, ductile coatings of this sort can only be obtained with high grade zinc having a cadmium content of less than 0.03 per cent. Lead in excess of 0.1 per cent is also considered by some galvanizers to be detrimental to the ductility of such coatings.

Figs. 39 to 42, inclusive, illustrate the microstructure of zinc coatings produced by the sherardizing, electrogalvanizing and zinc spray (Schoop) processes. The malleability of these coatings is of little importance since they are generally applied to the surface of the iron or steel object after all fabricating operations are completed. Their structure, however, may be worth studying if one is investigating the corrosion resistance of these types of zinc coatings.

The microstructures illustrated by Figs. 43 to 48, inclusive, are included to illustrate the great thickness of zinc coatings produced by the "hand dip" process. They also show the wide variety of forms taken by the zinc-iron alloy constituents which are probably due, in part at least, to the differences in composition of the steel base.

Acknowledgement.—The author is especially indebted to Mr. W. M. Peirce, Mr. A. C. Simmons and other members of the Metal Section staff, Research Division, New Jersey Zinc Co., whose kind assistance has made possible the preparation of this paper. He is also indebted to Mr. F. M. Crapo, Chief Engineer, Indiana Steel and Wire Co., who generously supplied a number of the galvanized wire samples.

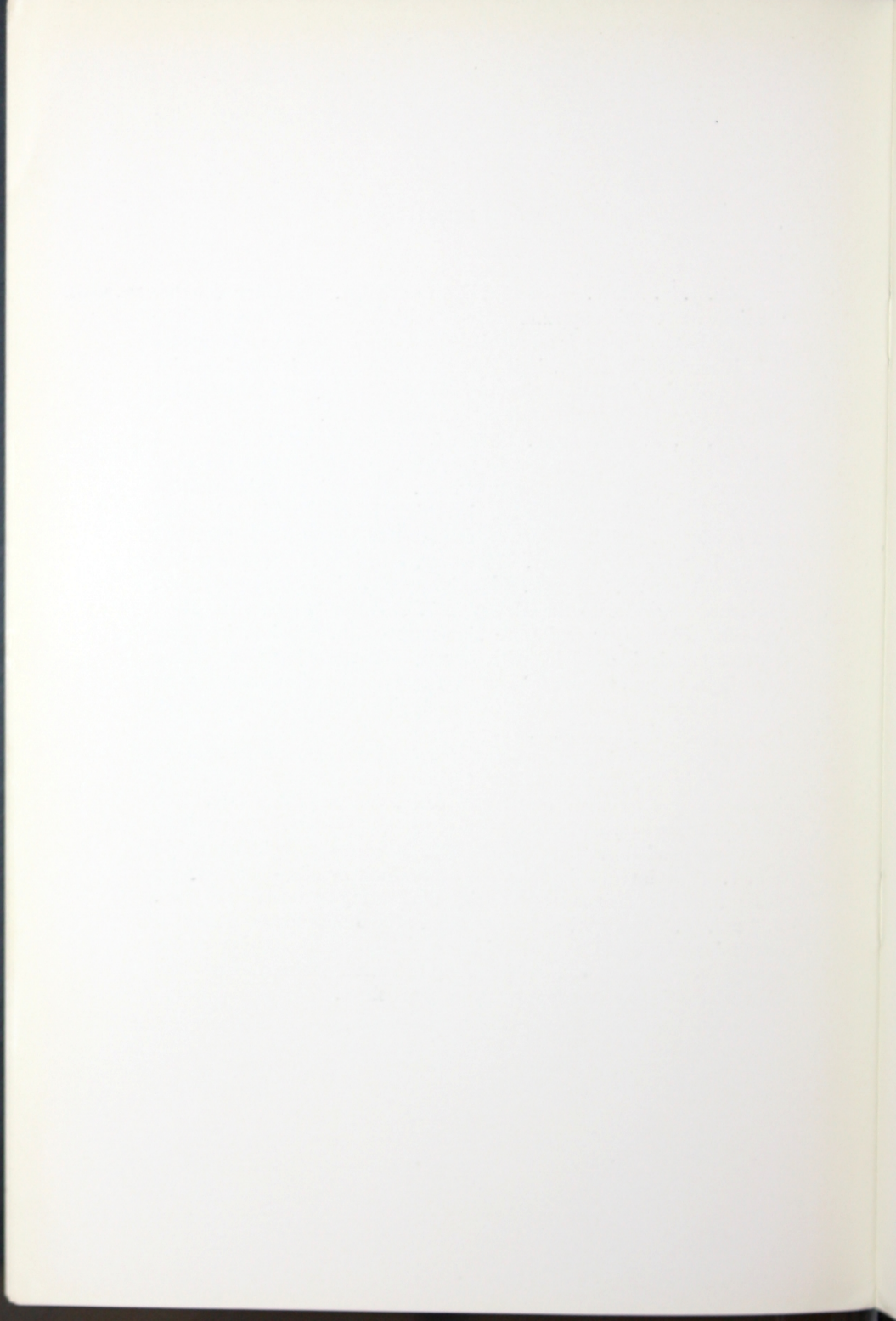
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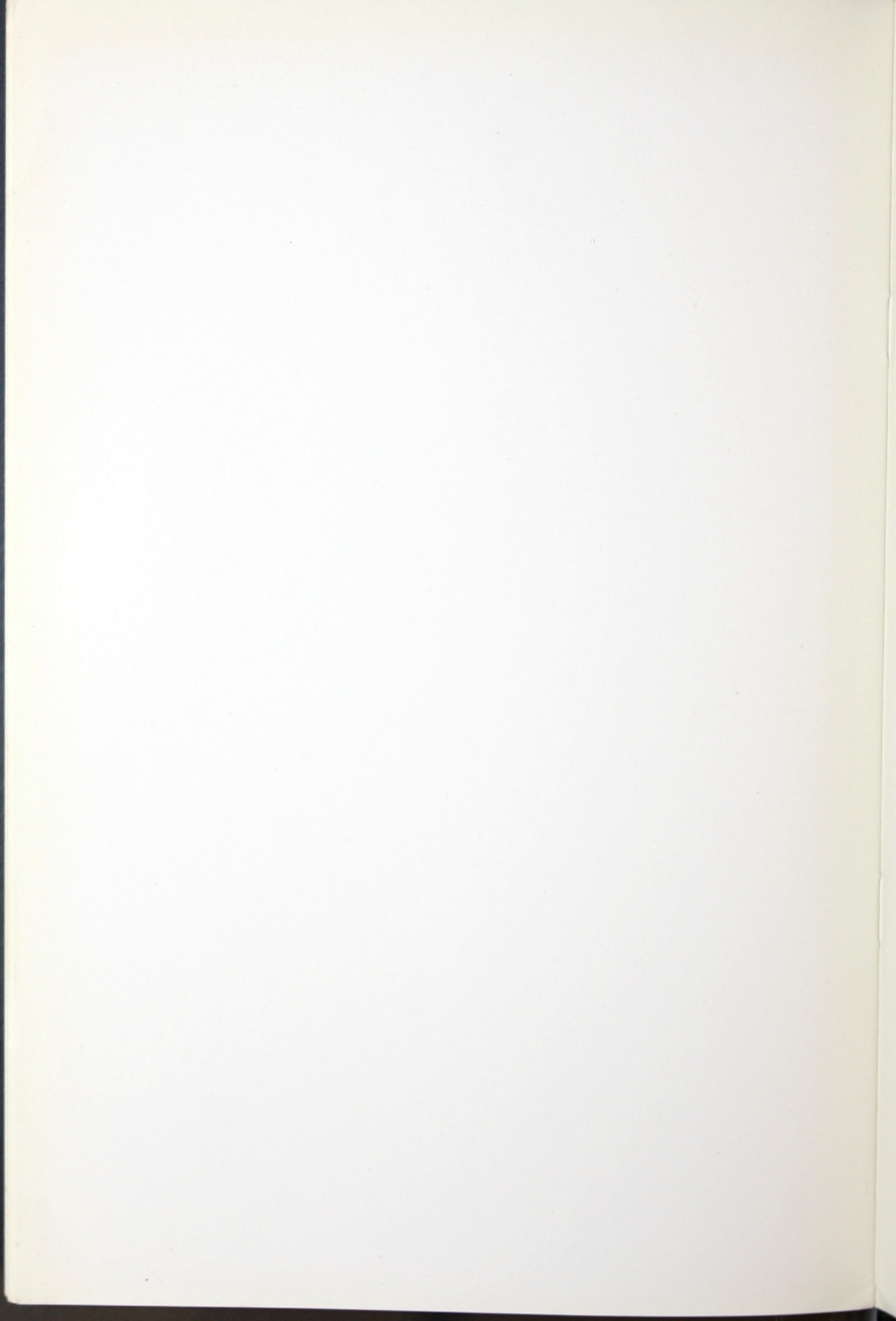
MR. J. T. KEMP.¹—I should like to ask Mr. Finkeldey whether the light band which appears on the steel base in a good many of the slides he has shown us, is an etching effect or a structure, and whether if it is an etching effect, it is due to any porosity in the iron-zinc compound alloy just above the steel base which might hold the etching solution after the samples were etched and washed. Mr. Kemp.

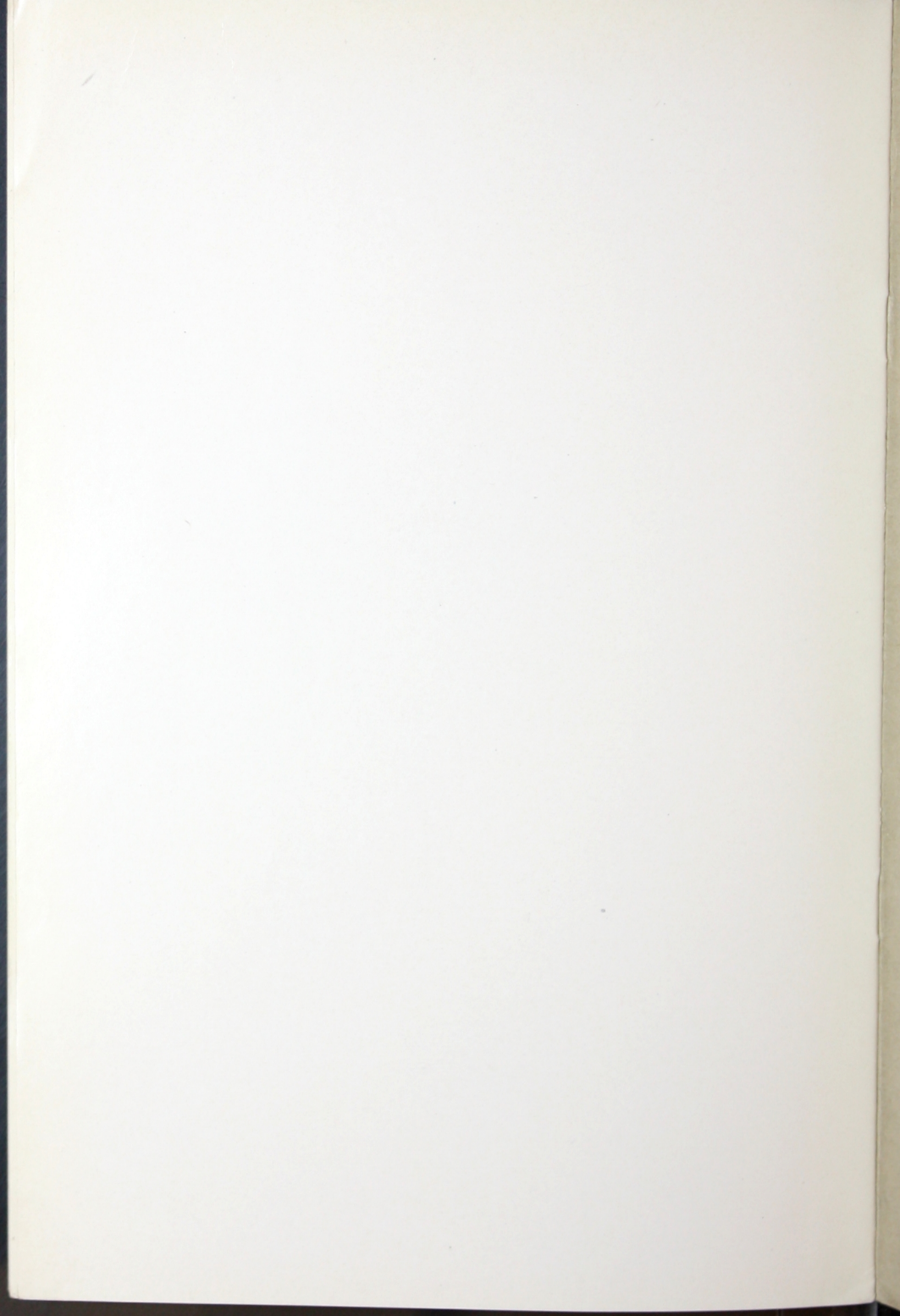
MR. W. H. FINKELDEY.—Where a zinc coating has been stressed as in the case of bent specimens there is a possibility that the alloy layers may separate from the steel base. In etching a micro-section from such a specimen, some solution might be retained in this crack and produce a staining effect on the steel which might be mistaken for an iron-zinc alloy layer. However, I believe in most cases the light band referred to by Mr. Kemp is one of the iron-zinc compounds. Unfortunately, due to the difference in elevation between the steel, iron-zinc alloy layer and pure zinc part of the coating, in many instances it is impossible, with our present polishing and etching technique, to prepare sections so that this narrow alloy band can be clearly seen and not mistaken for an etching effect or a shadow cast by the projecting steel base. Mr.
Finkeldey.

Figures 4 and 7 show structures having this narrow band of alloy. That this is actually a zinc alloy, and not a shadow or etching effect, is proved by the type of structure secured after heating a galvanized sheet in order to produce a growth of the alloy layers. This experiment was carried out with the sheet whose structure is illustrated in Fig. 7. The increase in thickness of the narrow alloy band can be easily traced at various stages during the heating by referring to Figs. 8, 9 and 10. The original narrow band of alloy has grown until it is approximately equal to the thickness of the entire coating and a second narrow band of iron-zinc alloy produced, which was not distinguishable in the structure of the original sheet.

¹ Technical Department, The American Brass Co., Waterbury, Conn.









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